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(54) Titre : REDUCTION ET OXYDATION DE METAUX DE TRANSITION, Y COMPRIS LES METAUX DE LA MINE DE PLATINE, SANS L'UTILISATION D'EAU REGALE

(54) Title: REDUCTION AND OXIDATION OF TRANSITION METALS, INCLUDING PLATINUM GROUP METALS, WITHOUT THE USE OF AQUA REGIA

(57) Abrégé/Abstract:
This invention has applications in the manufacturing of salts, analysis, mining, extraction and refining of transition metals, particularly Platinum Group Metals. A physical and electrochemical process in a compartmentalized electrolytic cell changes refractory transition metal compounds to readily soluble salts. The electrolytic cell has three compartments, a mixing chamber, a reduction chamber and an oxidation chamber. The mixing chamber contains the electrolyte, the substance processed, the reduction chamber and the oxidation chamber. In the reduction chamber, the negative electrode and the electrolyte produce a concentration of nascent hydrogen, which reduces the metals. In the oxidation chamber, the positive electrode and the electrolyte produce a concentration of nascent chlorine and oxidizing agents, which oxidize metals as soluble salts, or soluble complex ionic compounds. Convection currents or pumps circulate the substance processed through the various chambers.

ABSTRACT

This invention has applications in the manufacturing of salts, analysis, mining, extraction and refining of transition metals, particularly Platinum Group Metals. A physical and electrochemical process in a compartmentalized electrolytic cell changes refractory transition metal compounds to readily soluble salts. The electrolytic cell has three compartments, a mixing chamber, a reduction chamber and an oxidation chamber. The mixing chamber contains the electrolyte, the substance processed, the reduction chamber and the oxidation chamber. In the reduction chamber, the negative electrode and the electrolyte produce a concentration of nascent hydrogen, which reduces the metals. In the oxidation chamber, the positive electrode and the electrolyte produce a concentration of nascent chlorine and oxidizing agents, which oxidize metals as soluble salts, or soluble complex ionic compounds. Convection currents or pumps circulate the substance processed through the various chambers.

DESCRIPTION

TITLE OF THIS INVENTION:

Reduction and oxidation of transition metals, including Platinum Group Metals, without the use of Aqua Regia.

BACKGROUND:

Platinum occurs in the earth's crust far more abundantly than gold. The document, PLATINUM GROUP ELEMENT OCCURRENCES IN BRITISH COLUMBIA, produced by the Geological Survey Branch Ministry of Energy, Mines and Petroleum Resources of the Province of British Columbia, reports that specific locations in the Province of British Columbia contain Platinum Group Metals in amounts, up to and exceeding five kilograms per ton of black sands. Platinum Group Metals in black sands are invariable refractory oxides, insoluble in oxidizing acids, even in Aqua Regia. The Platinum Group Element oxides originate from volcanic and magmatic flows related to North American Plate and Pacific Plate tectonics.

The exploration and mining of the Platinum Group Metals in the North American Western Cordillera is not developed because of improper assay and extraction techniques. During the past twenty years, the inventor has actively prospected for precious metals in the Province of British Columbia. It has become obvious to the inventor that assay results, provided by conventionally adopted procedures of the industry are not commensurate with geological dictates and indicators.

The precious metal content of assayed rock samples is often not reflected in commercial analytical reporting, because of some of the following reasons:

1. Conventional assay reports are based on ICP-MS analysis of an Aqua Regia leachate. However, most Platinum Group Element oxides are insoluble in Aqua Regia.
2. Conventional fire assay procedures regularly prescribe soda ash and silicate fluxes. However when heated, Platinum Group Metals fuse and combine with sodium and with silicates; thus Platinum Group Metals are locked into the flux, instead of the collector.
3. Conventional fire assay procedures regularly use lead as a collector. However, the Platinum Group Metals are part of Group VIII of the Periodic Table; which do not readily fuse with lead; in fact the melting point of the Platinum Group Metals is generally higher than the boiling point of lead.
4. In the fire assay process, the approximate temperature is 1,000 degrees Celsius. However, at red heat, between 800 and 1200 degrees Celsius, Platinum Group Metals are in an environment, which promotes their oxidation not reduction.
5. Platinum dioxide has a density of 10.2. Lead has density of 11.4. The black platinum dioxide does not fuse with lead but floats on top of the liquid lead collector.
6. Alloys of gold and Platinum Group Metals occur jointly in minerals, which leads to many complications in the fire assay process and subsequently inaccurate analytical reports.

In addition to the improper analysis of the Platinum Group Metals, there are the following factors that prevent the development and mining of the Platinum Group Element resource:

1. Conventional extraction of naturally occurring transition metal oxides is highly specialized and often requires enormous amounts of energy.
2. Because of difficulties in extraction of Platinum Group Element oxides, companies of base and precious metal mining often focus on sulfides only and disregard the potential of precious metal oxides.

This invention helps resolve the problems in the analysis and extraction of transition metal oxides by transforming insoluble oxides to soluble salts. This is accomplished by improving the effectiveness of acids in an apparatus, hereinafter referred to as a "Redoxer." In a "Redoxer" hydrochloric acid, for example, is more effective than Aqua Regia. A "Redoxer" eliminates problems associated with Aqua Regia, and may relegate the use of Aqua Regia to antiquity.

A "Redoxer" does not use highly toxic materials, such as cyanide. It can be very effective with only three simple ingredients, muriatic acid (that is, 18% commercial grade hydrochloric acid), small amounts of concentrated sulfuric acid and sodium chloride, common cooking salt. A "Redoxer" requires relatively low amounts of energy. It operates on a relatively low voltage. A "Redoxer" does not require a prolonged leaching process. Depending on the application, a "Redoxer" may vary in size and shape.

The disclosure of this invention describes the physical and the electrochemical process in an apparatus, that the inventor used repeatedly to dissolve various ores and black sands, containing oxides of Platinum Group Metals, which otherwise are insoluble. In one specific case, the inventor heated and submerged the ore in various acids, even Aqua Regia, for a week; yet the ore remained virtually untouched. Then without pre-leaching this particular ore, the inventor dissolved in a "Redoxer" 190 of 250 grams of the refractory ore in approximately 30 minutes, by using an electrolyte of approximately one liter of muriatic acid (18% hydrochloric acid), 50 milliliters of concentrated sulfuric acid and 20 grams of sodium chloride. In a smaller "Redoxer" with a similar electrolyte, the inventor liquefied elemental gold, platinum, palladium and high concentrations of metal sulfides.

DESCRIPTION OF A SAMPLE APPARATUS WITH SPECIFIC DIMENSIONS:

The invention is an electrolytic cell comprising, at least the following three integral components:

1. A mixing chamber, in which the substance processed is blended with and partially suspended in the electrolyte,
2. At least one reduction chamber, which contains a negative electrode.
3. At least one oxidation chamber, which contains a positive electrode.

The mixing chamber is a circular glass test tube, 50 centimeters in height and 10 centimeters in diameter, comprising a convex bottom, and an open top that can be covered to control the escape of fumes. The mixing chamber contains the reduction chamber, the oxidation chamber, and the electrolyte, which is mixed with the substance processed.

The reduction and oxidation chambers are glass or corrosion and heat resistant plastic tubes that surround the electrodes. The reduction and oxidation chambers are 1.5 to 2.5 centimeters in diameter, at least 25 centimeters long. The tubes of these chambers are supported that they are adjacent and parallel to each other. The tubes are open at the bottom. The bottom of each tube is sufficiently removed from the bottom of the mixing chamber to facilitate convection currents. At start up of the process, the open-ended top of the reduction chamber is just above the level of the electrolyte.

The tube of the oxidation chamber is approximately double the length of the reduction tube and extends past the top of the mixing chamber; it helps support the reduction tube. Above the level of the electrolyte, the tube of the oxidation chamber serves as a conduit for the insulated electrical wires, leading to the electrodes. Approximately one centimeter above the level of the

electrolyte, the oxidation tube has ports in its side wall to facilitate convection currents in the electrolyte.

The distinction between reduction chamber and oxidation chamber is for explanatory purposes only. When in process, the functions of the reduction and oxidation tubes are interchangeable and can be reversed by reversing the electrical connections between the electrodes and the Direct Current voltage power supply. An occasional reversal of the polarity of the electrodes prevents a build-up of deposits on the electrodes, that may interfere with the efficiency of the process.

The electrodes are round carbon rods, at least 25 centimeters in length and 8 millimeters in diameter. The bottom of the electrodes extends through the reduction chamber and the oxidation chamber to the bottom of the mixing chamber. At start-up of the process, the top of each electrode is approximately two centimeters below the level of the electrolyte. The insulated electrical connections, leading to the electric power supply, are at the top of each electrode.

The electric power supply is reversible and variable 0 to 30 Volt D. C.; it has an output of at least 15 Amperes. The electric power supply serves two functions:

1. It heats the electrolyte and thus creates convection currents, which circulate the substance processed through the reduction chamber, the mixing chamber and the oxidation chamber.
2. The electric power supply facilitates the respective chemical reactions in the contact zone of the electrolyte and the electrodes.

Depending on the desired outcome, the electrolyte may be any organic or in-organic acid. In specific applications, the electrolyte may be a neutral solution of soluble salts with a pH factor of 7, sodium hydroxide or potassium hydroxide.

DESCRIPTION OF THE PHYSICAL PROCESS:

When the electrodes are energized by a Direct Current voltage supply the following process is initiated and sustained:

1. At start up, the greatest electrical resistance between the electrodes is at their top. The smallest electrical resistance between the electrodes is at their bottom. Therefore, there is a localized concentration of electrical current and heat at the very bottom of the reduction chamber and the oxidation chamber.
2. The heat, generated by the electrodes, rises to the top of the reduction and oxidation chamber and causes upward convection currents of the electrolyte in the reduction and oxidation chambers;
3. In addition to the heat, gases are released at the electrodes. The gases also rise to the top of the reduction and oxidation chambers and contribute to the convection currents.
4. When the temperature of the electrolyte reaches the boiling point, vigorously rising water vapors increase the strength of the convection currents.
5. The rising and boiling electrolyte is ejected out through the top of the reduction tube and passes back into the mixing chamber.
6. The rising and boiling electrolyte is ejected through the ports in the side wall of the oxidation tube and also passes back into the mixing chamber.
7. The substance ejected from top of the reduction and oxidation chambers quickly settles to the bottom of the mixing chamber, circulating and mixing the electrolyte with the substance processed, and forcing the substance processed to flow repeatedly through the reduction chamber and the oxidation chamber, thus bringing the substance processed into repeated and prolonged contact with the surface area of the electrodes.

8. Once the electrolyte has reached the boiling point, the rising froth and bubbles of the boiling electrodes establish electrical contact between the top of the electrodes, and thus increase the chemical efficiency of the electrodes.
9. Excessive amounts of the substance processed may require manual mixing, or provisions for slurry pumps, which assist the convection currents in the mixing and circulating of the electrolyte and the substance processed.

Depending on the density and the type of material of the substance processed, a "Redoxer," described in the sample apparatus above, can process up to 250 grams in less than one hour. For initial assay purposes, a test sample of 30 grams is normally sufficient. For the processing of larger amounts in large extraction plants, a circulatory system, induced by pumps, can eliminate the dependence on convection currents, reduce the operating temperature and reduce gaseous emissions of the electrolyte.

DESCRIPTION OF THE ELECTROCHEMICAL PROCESS:

In a hydrochloric electrolyte, a circuit of a Direct Current voltage power supply respectively causes the following reduction and oxidation at the negative and the positive electrodes.

At the negative electrode in the reduction chamber, a Direct Current voltage power supply creates a concentration of negatively charged electrons. This concentration of negatively charged electrons causes a concentration of positively charged hydrogen ions. The negatively charged electrons of the electrode reduce the hydrogen ions to non-molecular nascent hydrogen. The hydrogen reduces the metals of transition element oxides, sulfides and hydroxides suspended in the electrolyte to their atomic and metallic state.

At the positive electrode in the oxidation chamber, a Direct Current voltage power supply creates a deficiency of negatively charged electrons (that is, a positive charge). This deficiency of negatively charged electrons causes a concentration of negatively charged chlorine ions. The positively charged electrode oxidizes the chlorine ions to atomic non-molecular nascent chlorine (that is, a duplication of the of Aqua Regia effect, without the use of nitric acid). The nascent chlorine oxidizes the previously reduced metals and combines with the metals to form soluble salts and complex ionic compounds.

Nascent hydrogen and nascent chlorine form electrochemically in the contact zone of the electrodes and the electrolyte; but they exist as nascent hydrogen and nascent chlorine only momentarily. Therefore, the efficiency of a this process is directly proportional to the dimensions of the energized electrodes, the extent of their contact with the electrolyte, and the prolonged exposure of the substance processed to nascent hydrogen and nascent chlorine at the surface of the electrodes. This is accomplished in narrow, segregated reduction and oxidation chambers, by electrodes with a large surface area, and by the forceful flow of the electrolyte, which forcibly pushes the substance processed alongside the surface area of the electrodes. Increasing the strength and the flow of the electrolyte, the number and size of electrodes in their respective of reduction and oxidation chambers accelerates the process.

The dissolved transition metals can be filtered and separated from insoluble silicates and gangue material. The desired transition metals can be processed and refined.

CLAIMS

The embodiments and the process of the invention in which an exclusive property or privilege is claimed are defined as follows:

1. An apparatus of a compartmentalized electrolytic cell comprising at least one mixing chamber, at least one reduction chamber, at least one oxidation chamber, an electrolyte, at least one negative electrode, at least one positive electrode, provisions for electrical connections leading from the electrodes to a Direct Current voltage power supply, and provisions for the mixing and circulating of the electrolyte and the substance processed.

2. A process in an apparatus as defined in claim 1, in which, the electrodes, when energized by Direct Current voltage power supply, produce (A) the heat that generates circulating convection currents in the electrolyte, which transport the substance processed through at least one elongated, narrow reduction chamber and at least one elongated, narrow oxidation chamber, and exposes the substance processed to extended, elongated contact zones of the electrolyte and the respective electrodes, which produce (B) at the negative electrode, in the reduction chamber, nascent hydrogen, that reduces the metal of transition element oxides, hydroxides, sulfides, and produce (C) at the positive electrode, in the oxidation chamber, a concentration of the anions of the electrolyte, particularly chlorine ions, which are oxidized to nascent chlorine, that oxidizes transition metals, particularly the Platinum Group Metals, as soluble salts or soluble complex ionic compounds.

3. An apparatus and a process as defined in claim 1 and claim 2, in which said mixing chamber comprises an corrosion and heat resistant u-shaped container, which contains the substance processed, at least one reduction chamber, at least one oxidation chamber, an electrolyte, has provisions for

mixing and blending the electrolyte with the substance processed, and has provisions for circulating said mixture through said reduction chamber and said oxidation chamber by means of convection currents and/or by means of a circulatory system, provided by pumps or other mechanical means.

4. An apparatus and a process as defined in claim 1, claim 2 and claim 3, in which said reduction chamber and said oxidation chamber is a corrosion and heat resistant tube that is supported in the mixing chamber and is sufficiently removed from the bottom of the mixing chamber to facilitate the in-rush of convention currents of the electrolyte and the substance processed at the open-ended bottom of each tube.

5. An apparatus and a process as defined in claim 1, claim 2, claim 3 and claim 4, in which the open-ended top of said reduction chamber is just above the start up level of the electrolyte.

6. An apparatus and a process as defined in claim 1, claim 2, claim 3 and claim 4, in which the said tube, which serves as oxidation chamber, extends past the top of the mixing chamber; and which said tube, approximately one centimeter above the start up level of the electrolyte, has ports that facilitate the out-flow of the electrolyte and the substance processed back into the mixing chamber; and which said tube, above the level of the electrolyte, may contain the insulated wires leading to said positive electrode and said negative electrode.

7. An apparatus and a process as defined in claim 1, claim 2, claim 3, claim 4, claim 5 and claim 6, in which the distance between the surface area of the electrodes and the walls of the tubes containing the electrodes is kept at a minimum to maximize the contact of the substance processed with the surface

area of the electrodes, as convection currents and/or pumps force the substance processed through the reduction and oxidation chambers.

8. An apparatus and a process as defined in claim 1 and claim 2, in which said electrolyte comprises an organic acid, an inorganic acid, a mixture of acids and soluble salts, at least one ionized salt, aqueous sodium hydroxide or aqueous potassium hydroxide.

9. An apparatus and a process as defined in claim 1 and claim 2, in which said electrodes comprise carbon rods, which are approximately two or three centimeters shorter than the reduction chamber, and which protrude past the bottom of the reduction chamber and the oxidation chamber into the mixing chamber.